The SNS Magnetism Reflectometer

A Users Guide

May 27, 2011

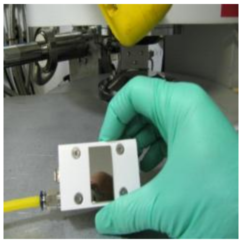
Contents

Sample Holders

The following sample holders are available:

Room Temperature

Maximum sample height: 49 mm
Sample fixation: vacuum



Low Temperature

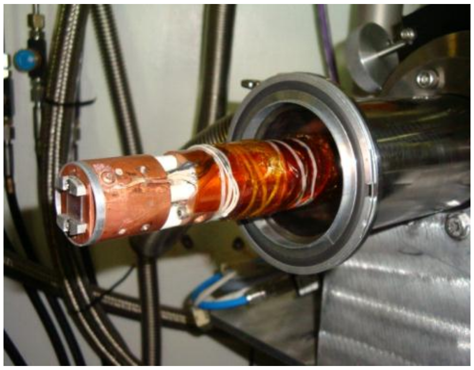
Sample Size: 20 mm x 20 mm maximum
Sample fixation:
Vacuum grease and absorbing flats for operation 5K < T < 300 K

Wire clips for operation 5K < T < 750 K and/or for axial rotation of the sample

Sample Environment

The following sample environment equipment is available:

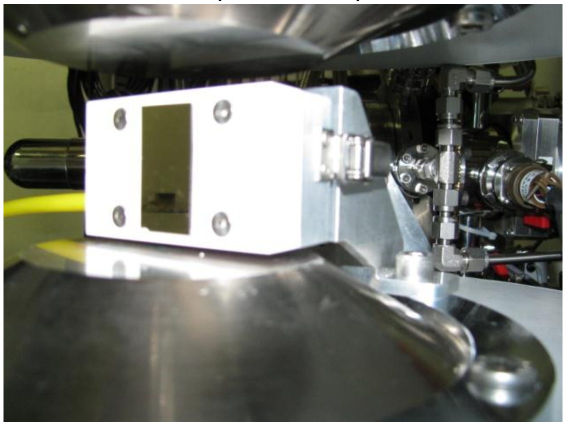
Displex



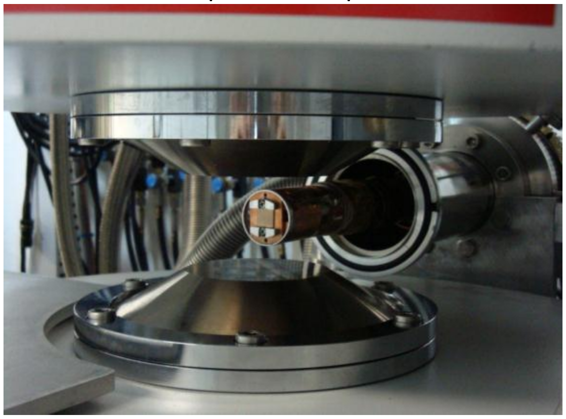
Operating Temperature Range: 5 Kelvin to 750 Kelvin
Axial Sample Rotation: 360 degrees

Electromagnet

Room Temperature Experiments



Low Temperature Experiments



Maximum magnetic field in conventional (displex) configuration (50 mm pole gap): 1.15 Tesla

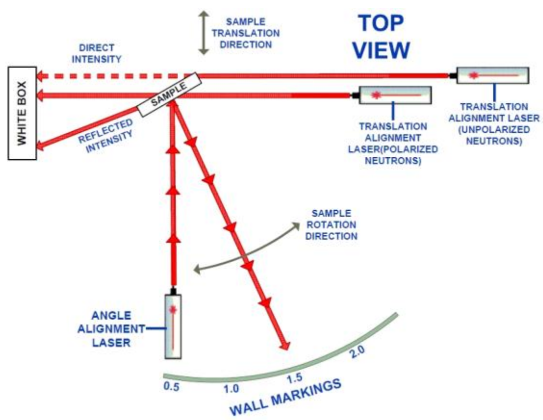
Other Magnet Pole Specifications

Pole Gap (mm)	Pole Face Diameter (mm)	Maximum Field (Tesla)
5	10	3.77
10	25	2.98
15	70	2.4
15	25	2.6
50	75	1.15
46	50	1.24
110	No Poles	0.565

Sample Alignment

Sample alignment consists of pre-alignment with the laser and alignment with neutrons.

1. Pre-alignment with Laser



1.1. Mount sample

1.1.1. Room temperature sample holder

1.1.1.1. Turn on vacuum pump
1.1.1.2. Position sample on sample holder so it covers vacuum hole in holder
1.1.1.3. Verify sample is retained by vacuum
1.1.1.4. Adjust sample position on holder if desired.
1.1.1.5. Attach holder to mount using retaining clips.

1.1.2. 300 K and below sample holder

1.1.2.1. Adjust borated aluminum flats for sample dimensions.
1.1.2.2. Apply thin, uniform layer of vacuum grease to sample holder and back of sample.
1.1.2.3. Place sample on holder, adjust position of sample and flats.
1.1.2.4. Using clean cotton swab, or finger protected by cotton glove or KimWipe, press sample onto holder so it is held by vacuum grease.
1.1.2.5. Verify vacuum grease retains sample to holder.
1.1.2.6. Thread sample holder to displex, making sure to include borated aluminum ring.
1.1.2.7. Use axial rotator to position sample holder so desired sample axis is straight up and down.
1.1.2.8. Rotate sample into center of pole pieces.

1.1.3. Above 300 K and axial rotation sample holder

1.1.3.1. Place sample in center of holder.
1.1.3.2. Position upper retaining clip at top of sample, as close to edge as possible to minimize scattering
1.1.3.3. Tighten screw on clip until clip holds sample in place

1.1.3.4. Repeat with lower clip

1.1.3.5. Thread sample holder to displex, making sure to include borated aluminum ring.

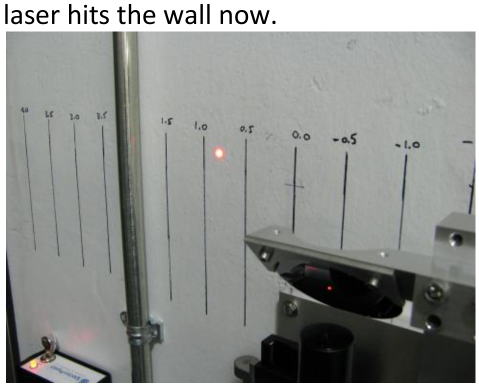
1.1.3.6. Use axial rotator to position sample holder so desired sample axis is straight up and down.

1.1.3.7. Rotate sample into center of pole pieces.

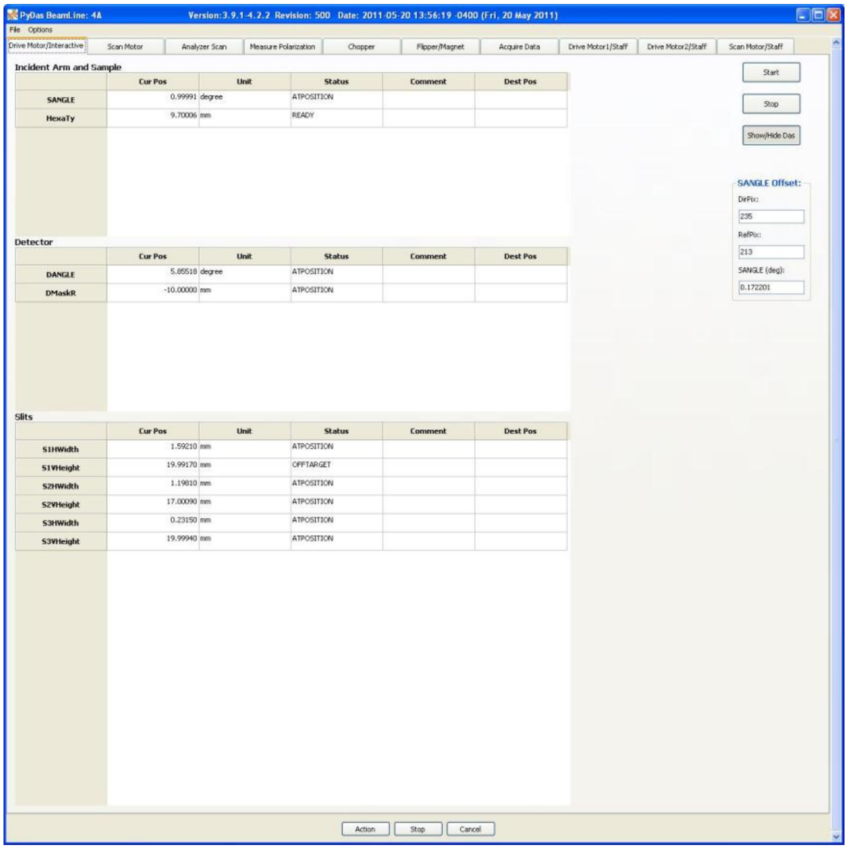
1.2. Turn on angle alignment laser

1.3. Rotate sample angle until laser reflection is approximately 0.5 degrees (use wall markings)

1.3.1. Look at where the laser hits the wall now.

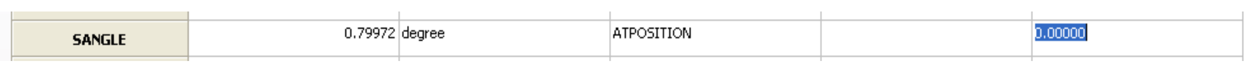


1.3.2. Use the *PyDAS* application (refer to PyDAS description) which is running on the computer terminal in the cave. Select the Drive Motor/Interactive tab.



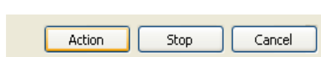
1.3.3.Calculate how far up or down the angle must move to hit the 0.5 degree wall marking.

1.3.4.In the *PyDAS* application find the line labeled *SANGLE*



1.3.5.Look at the value in the *Current Pos* field.

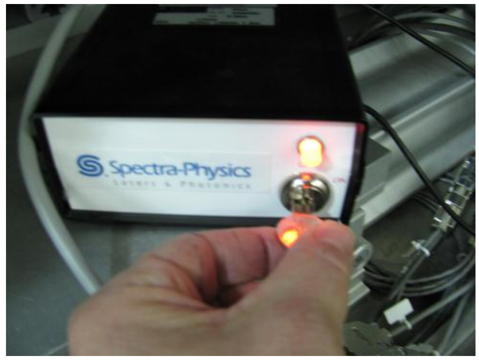
1.3.6.Calculate the value to enter in the *Dest Target* field to get the laser to hit around the 0.5 wall marking. Enter the value in the *Dest Pos* field. Press the *Action* button at the bottom of the *PyDAS* window. The sample angle will change. The system will go through several iterations. Once the status no longer says *MOVING* the system is finished.



1.3.7.Verify that laser hits wall at 0.5 degree mark.

1.4. Turn off angle alignment laser

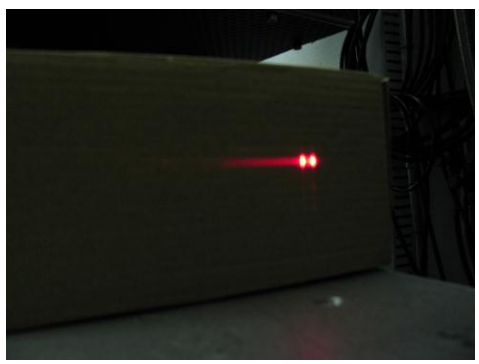
1.5. Turn on translation alignment laser



1.6. Place box in beam after sample position.
1.7. Translate sample until reflected laser intensity is maximized
   1.7.1. Go back to the *PyDAS* application
   1.7.2. In the *PyDAS* application find the line labeled *HexaTy*

HexaTy	1.55002 mm	READY		0.00000

   1.7.3. Repeat the process used previously: identify the *HexaTy* current position, enter a destination position, press action wait for the motion to finish.
   1.7.4. Check the intensity of the reflection.
   1.7.5. Repeat until reflected intensity is maximized.

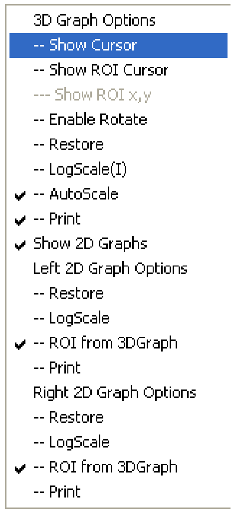


1.8. Turn off translation alignment laser.
1.9. Remove the box from the beam path.

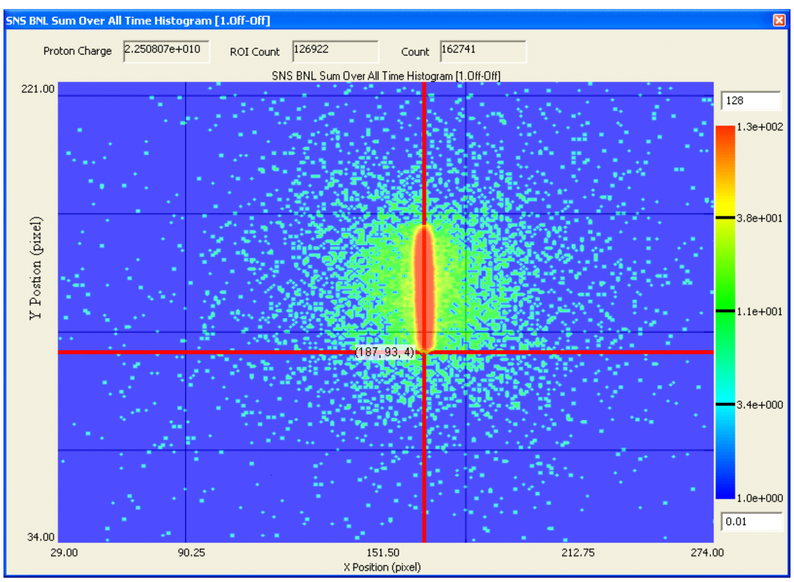
2. Alignment with Neutrons

2.1. Verify the position of the direct beam
   2.1.1. Open the secondary shutter.
   2.1.2. Switch to the *PyDAS* Window Interactive tab.
   2.1.3. Identify the current value for *DANGLE*.
   2.1.4. Set *DANGLE* to 4.00 degrees. This is the starting detector position we currently use for a low angle run, but the value could be anything.
   2.1.5. Set *DMaskR* to -5.00 mm. This will allow the direct beam to hit the detector.

2.1.6. Start data collection using the *Start* button in the *PyDAS Interactive* tab.
2.1.7. Switch to the window (display) labeled *1. Off-Off.*
2.1.8. If the cursor is not already visible right click on the right margin of the *1. Off-Off* display and select *Show Cursor*.

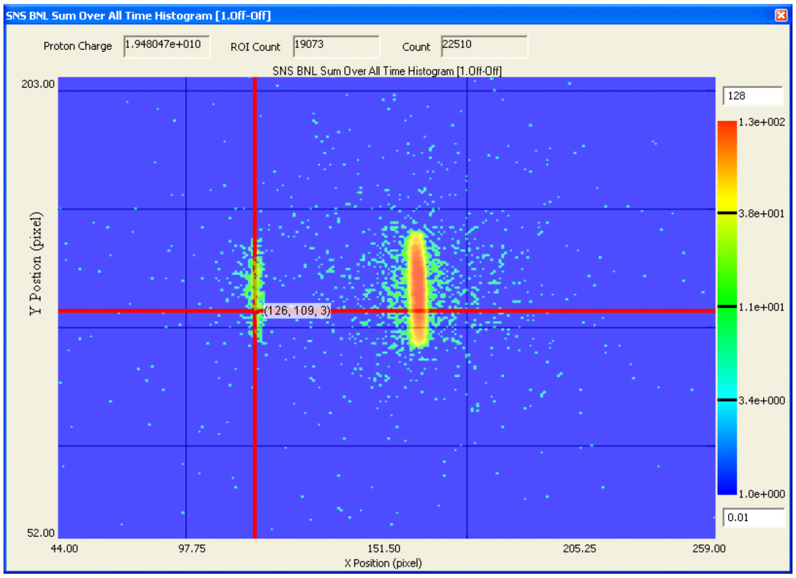


2.1.9. Observe the direct beam and position the cursor to identify the center pixel of the direct beam. The first number displayed next to the cursor is the x channel. Record that number.



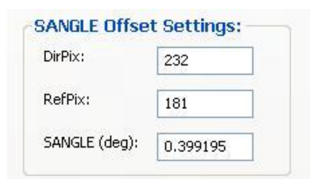
2.1.10. *Stop* the data collection by pressing the Stop button in the Interactive tab.
2.1.11. Move *DMaskR* to -45.00 mm to block the direct beam.

2.2. Calibrate the sample angle.
2.2.1. Start the data collection by pressing the *Start* button. In the Interactive tab.
2.2.2. Two discreet regions of intensity should be visible: the edge of the transmitted beam (on the right) and the reflected beam (on the left).



2.2.3.Grab the cursor with the mouse and position it at the center of the reflected signal.

2.2.4. At the right most side of the interactive tab is a box labeled *SANGLE Offset Settings* which contains two text fields: *DirPix* and *RefPix*.



2.2.5.Enter the pixel position of the direct beam in *DirPix* and the pixel position of the reflected beam in *RefPix*. The sample angle *SANGLE (deg)* will automatically be calculated.

2.2.6. Place the cursor in the text field which contains the sample angle and press the Enter key on the keyboard. The value of *Current Pos* for *SANGLE* in the main Interactive window should now be the angle just calculated.

2.2.7.*Stop* the run.

Data Collection

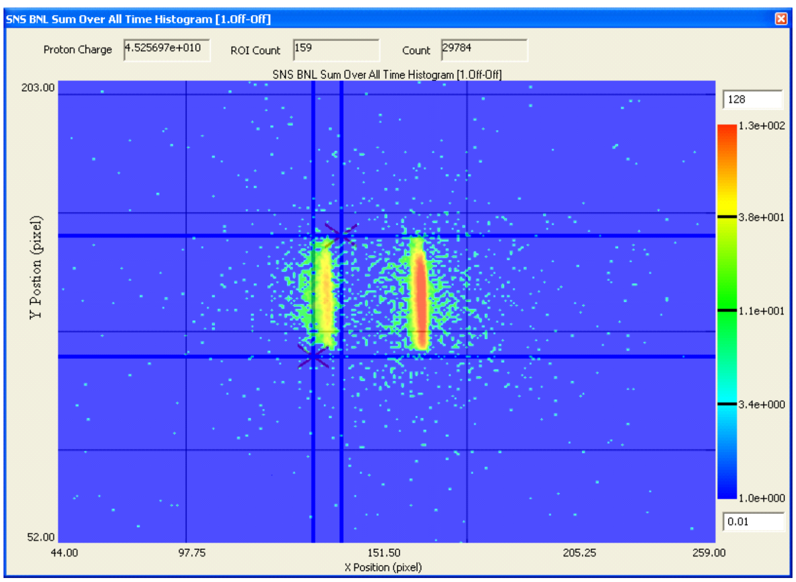
3. Final Sample Alignment

3.1. Room Temperature Sample Holder (No Displex)

3.1.1. Start data acquisition from the PyDAS Interactive tab.

3.1.2. If *the Region of Interest (ROI)* cursors are not already visible right click on the right margin of the *1. Off-Off* display and select *Show ROI Cursor*.

3.1.3. Grab each cursor with the mouse and position it until the *ROI* box just encompasses the reflected signal.



3.1.4. Switch to the *PyDAS* window *Interactive* tab. Find the entry labeled *S3HWidth* and set this width to 0.05 mm, using the same procedure used to change *SANGLE* and *HEXATY*.

3.1.5. Identify the current value of *HexaTy*.

3.1.6. Stop the data acquisition by using the *Stop* button in the *PyDAS Interactive* tab.

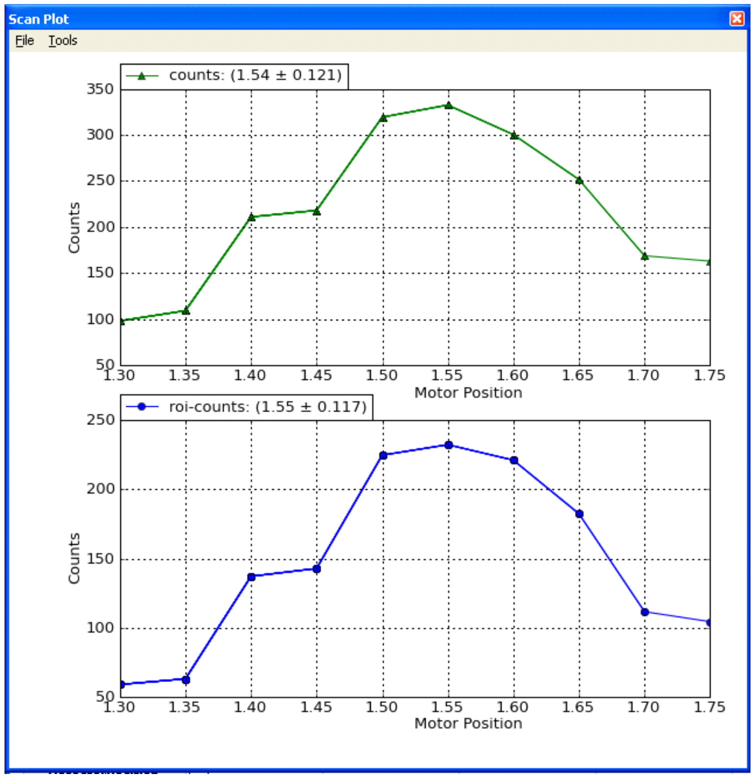
3.1.7. Switch to the *Scan Motor* tab.

3.1.8. Find the entry labeled *HexaTy*. Click the check box and enter scan limits to scan both sides of the current position. Enter the number of points to scan. Match the range of the scan and the number of points so that the step size is equal to the *S3Hwidth* value set previously.

3.1.9. Press the *Action* button

3.1.10. If the plot is not displayed, press the *Plot* button.

3.1.11. Once the scan is complete the plot will display the *HexaTy* position for maximum intensity (the bottom plot).



3.1.12. Switch to the *Interactive* tab and move *HexaTy* to the position of maximum intensity.

3.2. Displex Sample Holders

3.2.1. Cooling

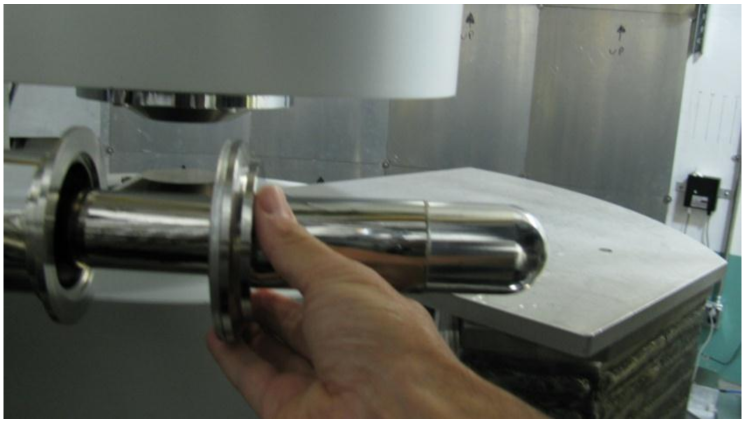
3.2.1.1. Enter the instrument cave.

3.2.1.2. Rotate the displex so that the cold finger is away from the magnet coils.

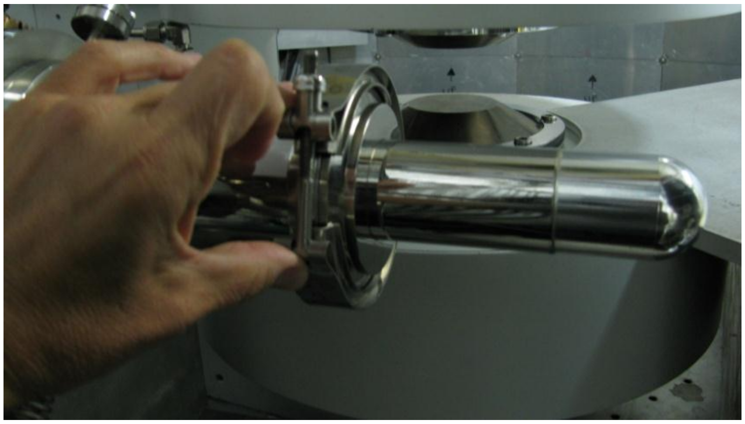
3.2.1.3. Carefully insert the Displex heat shield over the sample and cold finger.

3.2.1.4. Screw the heat shield on until it stops, but to not tighten.

3.2.1.5. Place the vacuum shroud and o-ring over the heat shield.



3.2.1.6. Fix the shroud in place with the retaining clamp and nut. Orient the clamp so the nut is on top, but make sure the excess thread does not interfere with the magnet coil.



3.2.1.7. Tighten the nut with the provided wrench.

3.2.1.8. Rotate the displex back until the rotation platform contacts the alignment screw. Do not over tighten.

3.2.1.9. Turn on the pump controller in the control hutch.

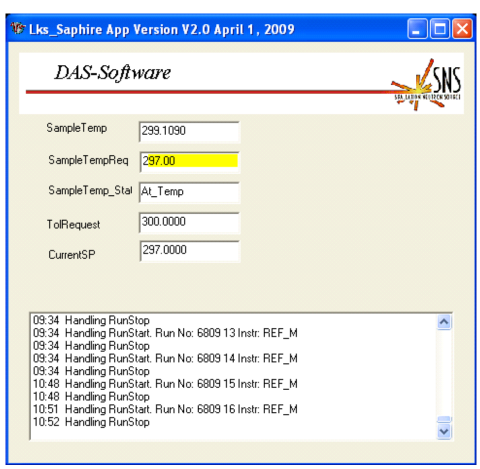
3.2.1.10. Observe the vacuum reading.

3.2.1.11. If the vacuum reading does not seem to progress downward in a reasonable amount of time check that the nut on the retaining ring is tight. Remove the vacuum shroud and check the o-ring for debris if necessary.

3.2.1.12. Once the pressure displayed on the pump controller is in the 10-4 range, turn on the Displex compressor using the knob on the compressor control panel.

3.2.1.13. Set the desired starting sample temperature.

3.2.1.14. Switch to the window labeled Lakeshore



3.2.1.15. Enter the desired temperature in the field labeled SampleTempReq.

3.2.1.16. Press the Enter key on the keyboard.

3.2.1.17. Allow one hour for the displex cold finger to contract completely. The cold finger will contract approximately 1 millimeter (at T < 100 K).

3.2.2. Cold Re-alignment

3.2.2.1. Verify the HexaTy alignment.

3.2.2.2. Identify the current HexaTy value.

3.2.2.3. Increase the value of HexaTy by ~1 millimeter to compensate for cold finger contraction.

3.2.2.4. Start data acquisition from the PyDAS Interactive tab.

3.2.2.5. Verify that the reflected beam is still visible. Some adjustment to the HexaTy value may be necessary.

3.2.2.6. Verify the sample angle.

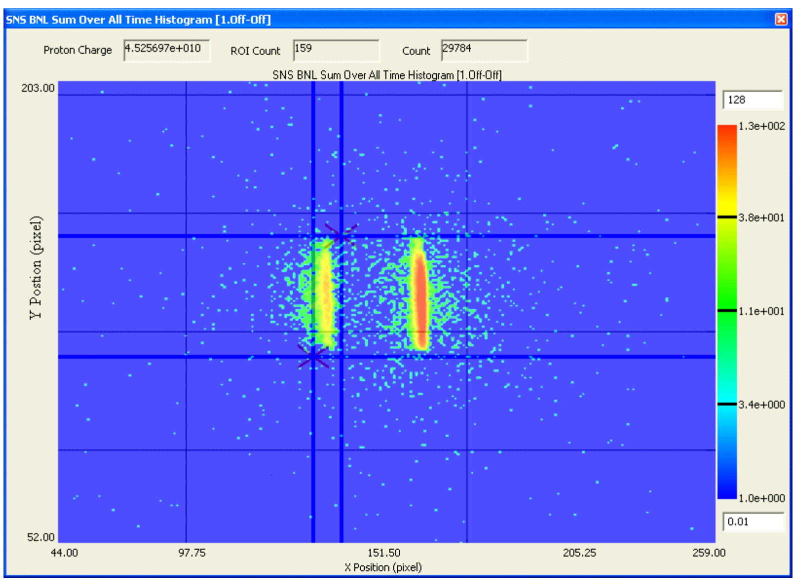
3.2.2.7. Verify the position of the reflected beam and calculate the sample angle, using the method described earlier.

3.2.2.8. Recalibrate the sample angle if necessary.

3.2.2.9. Alignment of the sample translation position to maximize the reflected intensity.

3.2.2.10. If *the Region of Interest (ROI)* cursors are not already visible right click on the right margin of the *1. Off-Off* display and select *Show ROI Cursor*.

3.2.2.11. Grab each cursor with the mouse and position it until the *ROI* box just encompasses the reflected signal.



3.2.2.12. Switch to the *PyDAS* window *Interactive* tab. Find the entry labeled *S3HWidth* and set this width to 0.05 mm, using the same procedure used to change *SANGLE* and *HEXATY*.

3.2.2.13. Identify the current value of *HexaTy*.

3.2.2.14. Stop the data acquisition by using the *Stop* button in the *PyDAS Interactive* tab.

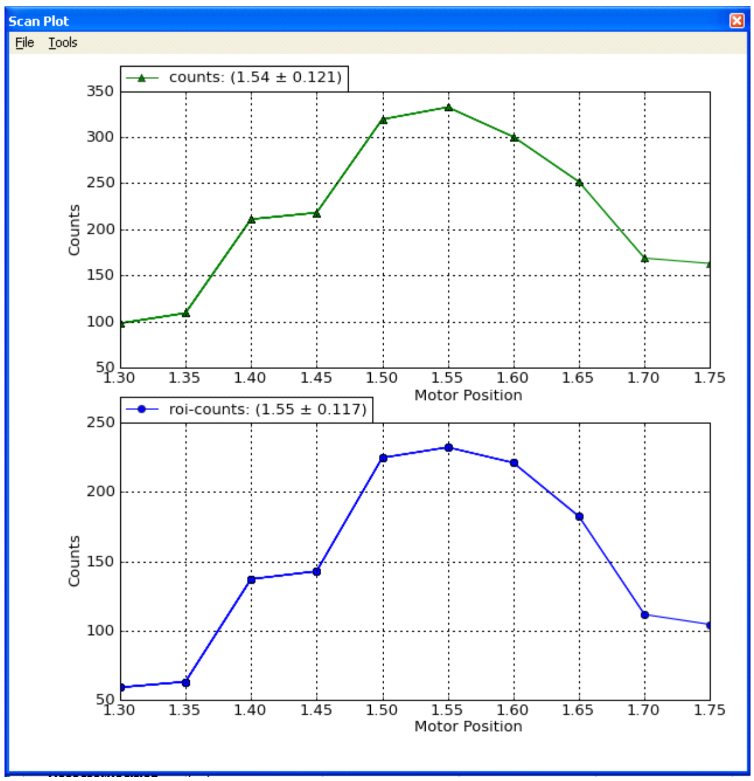
3.2.2.15. Switch to the *Scan Motor* tab.

3.2.2.16. Find the entry labeled *HexaTy*. Click the check box and enter scan limits to scan both sides of the current position. Enter the number of points to scan. Match the range of the scan and the number of points so that the step size is equal to the *S3Hwidth* value set previously.

3.2.2.17. Press the *Action* button

3.2.2.18. If the plot is not displayed, press the *Plot* button.

3.2.2.19. Once the scan is complete the plot will display the intensity as a function of the *HexaTy* position. Estimate the *HexaTy* for maximum intensity (use the bottom plot, which corresponds to the integral intensity in the ROI).

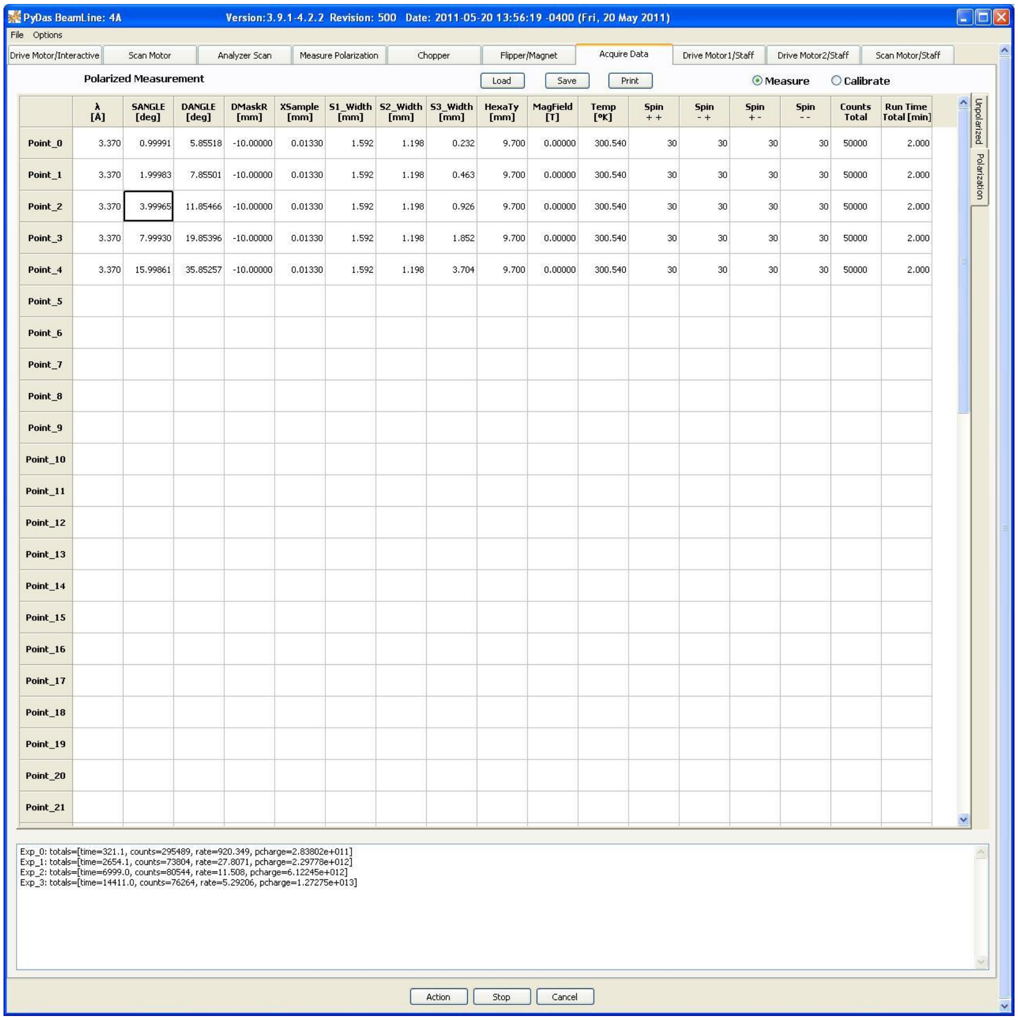


3.2.2.20. Switch to the *Interactive* tab and move *HexaTy* to the position of maximum intensity.

4. Acquiring Data

4.1. Select operating conditions (sample angle (*SANGLE*), detector angle (*DANGLE*), collimation (*S3HWidth*), temperature, magnetic field, etc.) for data collection. Consult beam line scientists.

4.2. Switch to the P*yDAS* Acquire Data tab. Note two side tabs: Conventional and Polarization. Select Polarization for polarized neutron reflectivity studies, and Convential for non-polarized neutron reflectivity studies.



4.3. Click the button at the top labeled Calibrate. Note that the default counting time for desired spin states when in calibration mode is thirty seconds.

4.4. Turn off any undesired spin states by right clicking on the column label and selecting state off.

4.5. On the first line labeled *Point_0* enter the operating conditions for the smallest sample angle to be run.
4.6. Enter the remaining angles to be run on subsequent lines.
4.7. Select the column labeled *DANGLE* by clicking on the shaded label field on top.
4.8. Right click *DANGLE label* field and select the *twoTheta* menu option to automatically calculate *DANGLE* and *S3_Width* settings for higher angles based on the *Point_0 SANGLE* setting.

4.9. Verify all settings and then click the button at the bottom labeled Action. PyDAS will automatically determine count rates for all angle settings and desired spin states. Final calculated count rates for each point and spin state will appear in the window at the bottom.
4.10. Note the position of the reflected signal with the *ROI* cursors displayed for all *SANGLE/DANGLE* pairs during calibration. If the pair values are correct the reflected signal should always be within the *ROI* cursors for all *SANGLE/DANGLE* pairs.
4.11. Wait for the calibration to finish.
4.12. PyDAS calculates optimum measuring times for each point based on the calculated count rates. These times can be adjusted if desired. PyDAS will calculate total counts for each point based on the calculated count rates.
4.13. Click the button at the top labeled Measure.
4.14. Verify that the *Point_0* current settings for *SANGLE*, *DANGLE* and *S3_Width* are the lowest angle settings

4.15. Select the column labeled *DANGLE* by clicking on the shaded label field on top.
4.16. Right click *DANGLE label* field and select the *twoTheta* menu option to automatically calculate *DANGLE* settings for higher angles based on the *Point_0 SANGLE* setting.

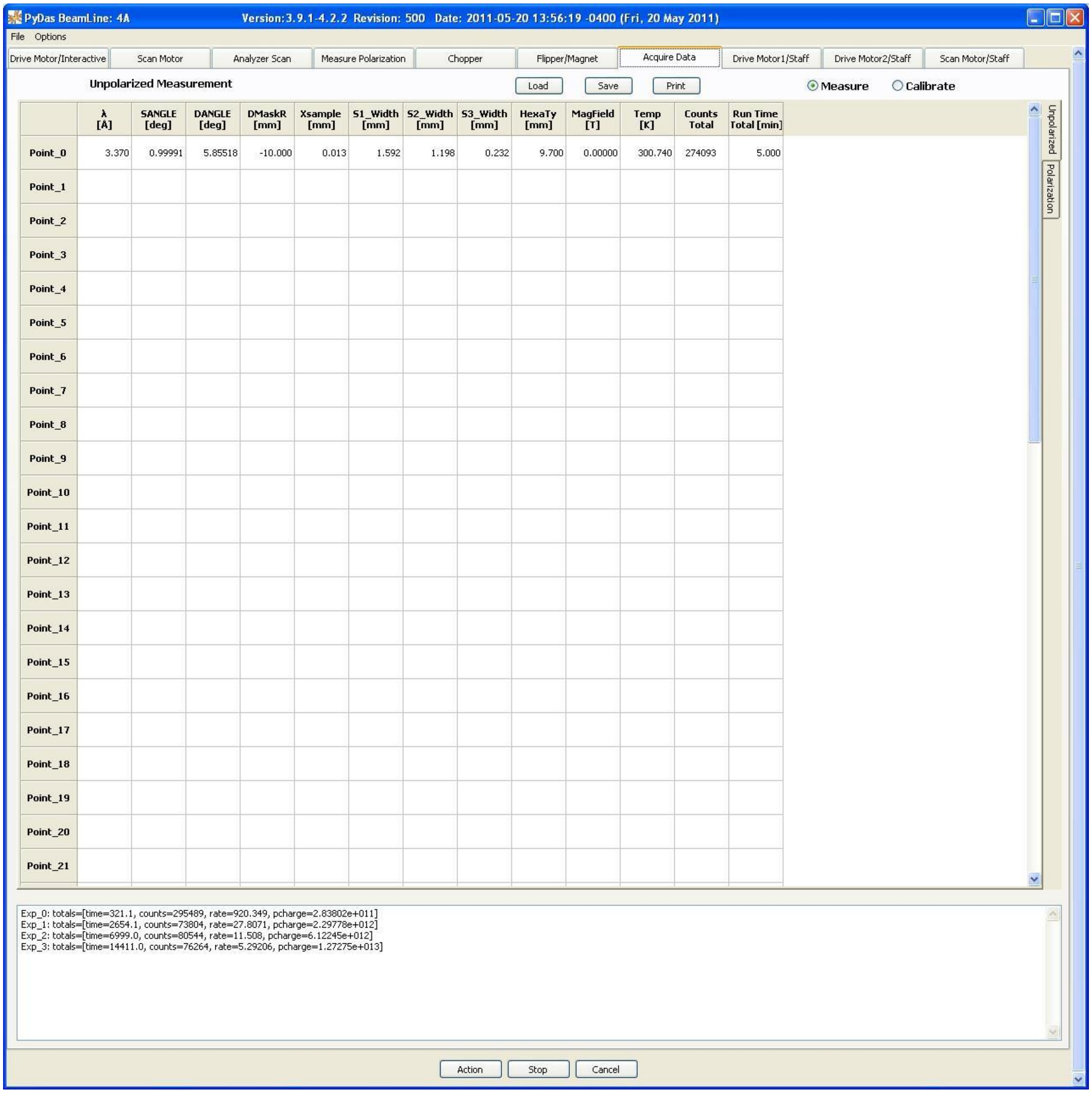
4.17. Save this configuration of runs, if desired by clicking the button labeled *Save* at the top of the window.
4.18. Verify all settings
4.19. Click the *Action* button to begin data collection.

5. Transmission Run

5.1. Transmission runs (runs with no sample in the beam) are required for data analysis and may be performed before or after sample runs.

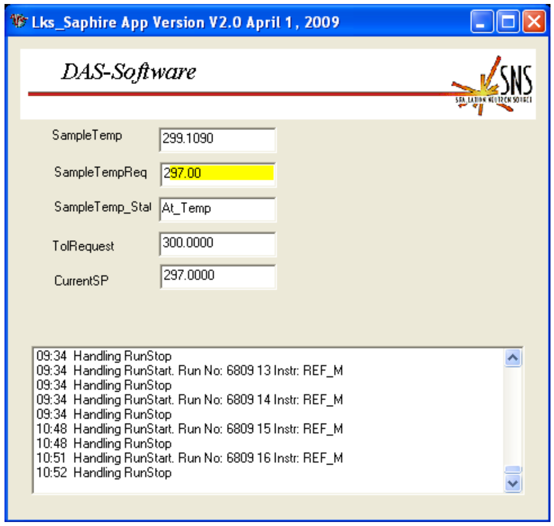
5.2. Set *DMaskR* to -5.00 mm to allow the direct beam to hit the detector.
5.3. Open the secondary shutter.
5.4. If necessary, move the sample out of the neutron beam by translating *HexaTy* 10 millimeters in the negative direction using the *Interactive* tab in the *PyDAS* window.
5.5. Set the desired collimation for *S3HWidth*.

5.6. Start collecting data and verify that no reflection is present.
5.7. Stop collecting data.
5.8. Switch to the *Acquire Data* tab in the *PyDAS* window.
5.9. Select the side tab labeled *Conventional*.



5.10. Set up one transmission run.
5.11. Insure that all parameters are correct.
5.12. Insure that the button labeled Measure is selected.
5.13. Verify both flippers are set to OFF.
5.14. Set the value for *Temp* to the current temperature.
5.15. Switch to the *Lks_Saphire App* window.
5.16. Observe the *SampleTemp* field value.

5.17. Enter this value into the *Temp* field in the *PyDAS Acquire Data Conventional* window.



5.18. If necessary widen the tolerance window by increasing the value in the *TolRequest*.
5.19. The standard *TolRequest* for normal data collection is 5.00 Kelvin.
5.20. Increased to 300 K (if desired) so that transmission run will collect regardless of sample temperature (useful if transmission run is collected during system warm up).
5.21. Set count units value to infinity if run is to be stopped manually.
5.22. Click *Action* to begin collecting transmission run data.
5.23. When transmission run is completed click *Stop* button.
5.24. Change *TolRequest* back to 5.00 K
5.25. Translate *HexaTy* to move sample back into beam.
5.26. Enter instrument cave and move direct beam shutter back in front of detector.

6. Sample Changing

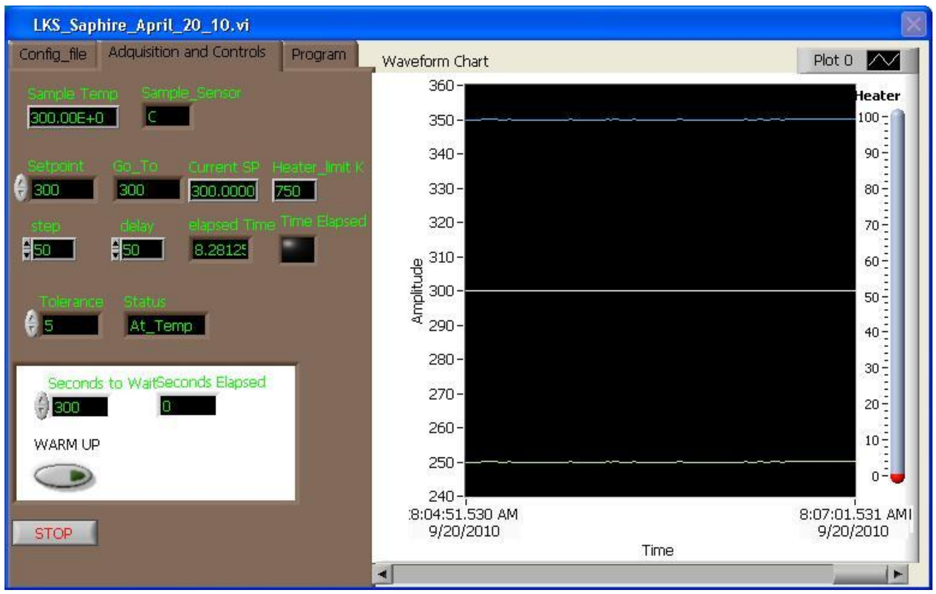
6.1. Room Temperature

6.1.1. Turn off the vacuum pump.
6.1.2. Perform a sample survey.
6.1.3. Slowly slide your sample away from the center of the sample holder until it clears the vacuum port.
6.1.4. Remove the sample.
6.1.5. Use the room temperature sample mounting procedure discussed earlier to mount the next sample.

6.2. Displex

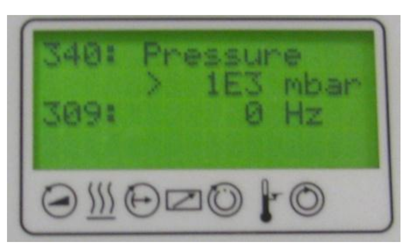
6.2.1. Verify the run is **stopped**.
6.2.2. Turn off the **Bruker Magnet**.
6.2.3. Turn **off** the displex compressor.

6.2.4.Find the *LKS_Saphire* application on the *Sample Environment* computer.



6.2.5.Press the button at the bottom labeled *Warm Up*. The green light on the button will turn on.

6.2.6.**Wait** for **one and a half hours** while the displex cold finder warms up to room temperature.
6.2.7.Press the power button on the pump controller to begin the venting process.
6.2.8.Wait approximately five minutes.
6.2.9.Verify the turbo pump speed is zero and the displex is at atmospheric pressure.



6.2.10. Go back to the **LSK_Saphire** Application on the **sample environment computer** and press the **Warm Up** button again to turn off the warm up cycle. The green light should no longer be illuminated.
6.2.11. Verify the electromagnet is **off**.
6.2.12. Rotate the displex out of the magnet poles.
6.2.13. Remove the displex shroud.
6.2.14. Remove the displex heat shield.
6.2.15. Perform a sample survey.
6.2.16. Remove the sample holder from the cold finger.
6.2.17. Remove the sample from the sample holder.
6.2.18. Use the low temperature sample mounting procedure discussed earlier to mount the next sample.